National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1974c

Organics in Mussel Tissue (*Mytilus edulis*)

This Standard Reference Material (SRM) is a frozen mussel tissue homogenate intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and polybrominated diphenyl ether (PBDE) congeners in marine bivalve mollusk tissue and similar matrices. All of the constituents for which certified and reference values are provided in SRM 1974c were naturally present in the tissue material before processing. A unit of SRM 1974c consists of five jars each containing approximately 10 g (wet basis) of frozen tissue homogenate.

Certified Mass Fraction Values: Certified mass fraction values for 22 PAHs, 38 PCB congeners, 11 chlorinated pesticides, and 5 PBDE congeners are provided in Tables 1 to 4. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. The certified values are based on the agreement of results obtained at NIST using multiple analytical techniques. The measurand is the total mass fraction of each analyte listed in Tables 1 to 4. Metrological traceability is to the SI unit for mass (expressed as micrograms per kilogram).

Reference Mass Fraction Values: Reference mass fraction values are provided in Tables 5 to 7 for an additional 18 PAHs, 14 PCB congeners, and 2 chlorinated pesticides, respectively. A NIST reference value is a non-certified value that is the best estimate of the true value; however, the value does not meet the NIST criteria for certification and is provided with an associated uncertainty that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1]. The measurand is the mass fraction of each analyte listed in Tables 5 to 7 as determined by the methods used. Metrological traceability is to the SI unit for mass (expressed as micrograms per kilogram).

Expiration of Certification: The certification of **SRM 1974c** is valid, within the measurement uncertainty specified, until **30 September 2022**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of technical measurements leading to certification were performed by M.M. Schantz and L.C. Sander of the NIST Chemical Sciences Division.

Preparation of the material was performed by G. Ballihaut, P.R. Becker, W.C. Davis, M.B. Ellisor, J. Hoguet, A.J. Moors, B.J. Porter, R.S. Pugh, L.B. Rust, and J.M. Yordy of the NIST Chemical Sciences Division.

Analytical measurements were performed by M.M. Schantz and S.S. Van der Pol of the NIST Chemical Sciences Division.

Statistical analyses of the certification data were performed by N.A. Heckert and A.L. Pintar of the NIST Statistical Engineering Division.

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Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

NOTICE TO USERS: SRM 1974c IS INTENDED FOR LABORATORY USE ONLY, NOT FOR HUMAN CONSUMPTION.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Storage: The SRM should be stored at –80 °C (or lower). Extended storage at temperatures of –25 °C or higher, or if allowed to warm, the tissue homogenate will lose its powder-like form.

Handling: For the handling of this material during sample preparation, the following procedures and precautions are recommended:
- If weighing relatively large quantities (≥3 g), remove a portion from the jar and reweigh the jar to determine the weight of the subsample. (Avoid heavy frost buildup by handling the jars rapidly and wiping them prior to weighing.)
- For weighing smaller quantities, transfer subsamples to a pre-cooled thick-walled glass container rather than a thin-walled plastic container to minimize heat transfer to the sample.
- If possible, use a cold work space, e.g., an insulated container with dry ice or liquid nitrogen coolant on the bottom and pre-cooled implements, such as Teflon-coated spatulas, for transferring the powder.
- If the material has been previously thawed and is no longer powder-like, allow the sample to completely thaw, stir well, and use the contents of the entire jar for analysis.

Use: Subsamples of this SRM for analysis (minimum of 3 g) should be withdrawn from the jar immediately after opening and used without delay for the certified values listed in Tables 1 to 4 to be valid within the stated uncertainties. The mass fractions of constituents in SRM 1974c are reported on both a wet-mass and a dry-mass basis for user convenience. The SRM tissue homogenate, as received, contains approximately 90 % moisture. A separate subsample of the SRM should be removed from the jar at the time of analysis and dried to determine the concentration on a dry-mass basis (see "Conversion to Dry-Mass Basis").

PREPARATION AND ANALYSIS[1]

Sample Collection and Preparation: The mussels (*Mytilus edulis*) used for the preparation of SRM 1974c were collected in Dorchester Bay, MA in 2004 by TDI-Brooks International, College Station, TX. The mussels were frozen and delivered to NIST (Hollings Marine Laboratory, Charleston, SC) where they were stored in a liquid nitrogen (LN_2) vapor-phase freezer at –150 °C. For processing, the mussels were allowed to warm to approximately 0 °C, shells were opened, and the tissue removed using titanium knives. Approximately 70 kg of mussel tissue was stored in Teflon bags in an LN_2 vapor-phase freezer (–150 °C) until homogenization. For homogenization, the frozen mussel material was removed from the Teflon bags, placed in a pre-frozen Teflon smasher, and crushed into smaller pieces using a manual smashing device and/or a compressed-air smashing device. The frozen, crushed mussel material was then immediately placed back in an LN_2 vapor-phase freezer (–150 °C) and divided among four stainless steel buckets within the freezer. The Palla VM-KT Vibrating Cryomill (KHD Humboldt Wedag GmbH, Cologne, Germany) was cooled allowing LN_2 to flow through the mill until a temperature of –180 °C was reached. The LN_2 was shut off and the crushed mussel tissue from all 4 buckets was processed through the cryomill until a fresh, frozen powder was created. This procedure was repeated four times prior to bottling to ensure the mussel material was completely blended. Subsamples (approximately 10 g) of the frozen mussel powder homogenate were aliquoted into cleaned, pre-cooled glass jars within an LN_2 vapor-phase freezer (–150 °C) and the glass jars were then stored in –80 °C upright mechanical freezers.

Conversion to Dry-Mass Basis: Sixteen samples were analyzed for moisture using an automated moisture/solids microwave analysis system (CEM, Matthews, NC). Each sample was approximately 1 g of material; the automated moisture determination temperature maximum was set to 105 °C and the power was set to 100 %. A sample was determined to have reached dry mass when the mass of the sample had not changed more than 0.1 mg in 10 s. The moisture content at the time of the certification analyses was 89.75 % ± 0.08 % (expanded uncertainty at a 95 % confidence level for 16 samples with a standard deviation of 0.0015). Analytical results for the constituents were determined on a wet-mass basis and then converted to a dry-mass basis by dividing by the conversion factor of 0.1025 (grams dry mass per gram wet mass). The uncertainty component for the conversion factor obtained from the

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[1] Certain commercial equipment, instrumentation, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

moisture measurements is incorporated in the uncertainties of the certified and reference values using the methods of reference 6, reported on a dry-mass basis, that are provided in this certificate.

PAHs, PCBs, Chlorinated Pesticides, and PBDEs: Value assignments of the PAHs, PCBs, chlorinated pesticides, and PBDEs in SRM 1974c consisted of combining results from analyses using various combinations of different extraction techniques, cleanup/isolation procedures, and chromatographic separation and detection techniques. Two sets of gas chromatography/mass spectrometry (GC/MS) analysis methods, designated as GC/MS (I) and GC/MS (II), were used at NIST.

For GC/MS (I) analyses, duplicate test portions of approximately 3 g from each of 10 jars of SRM 1974c were mixed with diatomaceous earth (Hydromatrix, Restek, Bellefonte, PA) in glass extraction thimbles. The mixtures were extracted using Soxhlet extraction with hexane:acetone (1:1 volume fraction) for 20 h. The extract was fractionated using two aminopropyl solid-phase extraction (SPE) columns to isolate the fraction of interest. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. $\times$ 60 m fused silica capillary column with a 50 % (mole fraction) phenyl methylpolysiloxane phase (0.25 μm film thickness; DB-17MS, Agilent Technologies, Wilmington, DE). The PAHs, PCBs, and pesticides were analyzed on the DB-17MS column using electron impact MS (EI-MS), method GC/MS (Ia). The PBDEs were analyzed on a 0.25 mm $\times$ 15 m fused silica capillary column containing a 5 % phenyl methylsubstituted polysiloxane phase (Restek), 0.25 μm film thickness using negative chemical ionization MS (NCI-MS), method GC/MS (Ib).

For the GC/MS (II) analyses, a 9 g sample from each of six jars was extracted using pressurized-fluid extraction (PFE) with dichloromethane (DCM). The fraction of interest was first isolated using an alumina (5 % deactivated) SPE column. Size exclusion chromatography (SEC) on a divinylbenzene-polystyrene column (10 μm particle size, 10 nm (100 Å) pore size, 7.5 mm $\times$ 300 mm i.d. PLGel column, Polymer Labs, Inc., Amherst, MA) was used to remove the majority of the remaining lipid and biogenic material. The processed extract was then analyzed by GC/MS using a 0.18 mm i.d. $\times$ 30 m fused silica capillary column with a low-bleed, low-polarity phase (0.18 μm film thickness; DB-XLB, Agilent Technologies, Wilmington, DE). The PAHs, PCBs, PBDEs, and certain pesticides were analyzed on the DB-XLB column using EI-MS, method GC/MS (IIa). The remaining pesticides were analyzed on the same capillary column using NCI-MS, method GC/MS (IIb). For the methods described above, selected perdeuterated PAHs, carbon-13 labeled PCB congeners, chlorinated pesticides, and PBDE congeners, and fluorinated PBDE congeners were added to the mussel tissue prior to extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs, PCBs, Chlorinated Pesticides, and PBDEs: The homogeneity of SRM 1974c was assessed by analyzing duplicate test portions of 3 g from 10 jars selected by stratified random sampling. Test portions were processed and analyzed as described above for GC/MS (I). No differences among jars were observed for the PAHs, PCBs, chlorinated pesticides, or PBDEs for a 3 g test portion size.

Table 1. Certified Mass Fraction Values for Selected PAHs in SRM 1974c

	Mass Fraction[a,b,c] (µg/kg)						k
	Wet-Mass Basis			Dry-Mass Basis			
Fluorene	2.31	±	0.04	22.6	±	0.4	1.97
Dibenzothiophene	1.53	±	0.02	15.0	±	0.2	1.99
Phenanthrene	19.6	±	0.4	191	±	4	1.96
Anthracene	1.17	±	0.08	11.4	±	0.8	1.97
1-Methylphenanthrene	3.07	±	0.11	30.0	±	1.1	1.97
2-Methylphenanthrene	4.56	±	0.04	44.5	±	0.5	1.97
3-Methylphenanthrene	4.09	±	0.03	39.9	±	0.4	1.97
9-Methylphenanthrene	2.46	±	0.02	24.0	±	0.3	1.97
2-Methylanthracene	0.951	±	0.007	9.3	±	0.1	1.97
Fluoranthene	45.3	±	0.8	442	±	9	1.97
Pyrene	23.9	±	1.6	233	±	15	1.97
Benzo[ghi]fluoranthene	3.03	±	0.09	29.5	±	0.9	1.96
Benzo[c]phenanthrene	1.99	±	0.04	19.4	±	0.4	1.97
Benz[a]anthracene	5.69	±	0.11	55.5	±	1.1	1.96
Benzo[k]fluoranthene	2.75	±	0.02	26.8	±	0.3	2.04
Benzo[a]fluoranthene	0.543	±	0.006	5.30	±	0.07	1.97
Benzo[e]pyrene	7.33	±	0.05	71.6	±	0.7	1.98
Benzo[a]pyrene	2.32	±	0.03	22.6	±	0.3	1.96
Perylene	0.560	±	0.022	5.46	±	0.22	1.97
Benzo[ghi]perylene	2.82	±	0.05	27.6	±	0.5	1.97
Benzo[b]chrysene	0.694	±	0.013	6.77	±	0.13	1.97
Picene	1.36	±	0.08	13.2	±	0.8	1.97

[a] Mass fractions are reported on both wet- and dry-mass basis; material as received contains 89.75 % ± 0.08 % (95 % confidence level) water.

[b] The certified value reported on a wet-mass basis is a weighted mean of average mass fractions, with one average each from two analytical methods [3,4]. The expanded uncertainty is the half width of a symmetric 95 % parametric bootstrap confidence interval [5], which is consistent with the ISO Guide [6,7]. The effective coverage factor k is included in the table for each PAH.

[c] GC/MS (Ia) using SPE clean-up followed by analysis on a DB-17MS column using EI-MS and GC/MS (IIa) using SPE and SEC clean-up followed by analysis on a DB-XLB column using EI-MS.

Table 2. Certified Mass Fraction Values for Selected PCB Congeners[a] in SRM 1974c

			Mass Fraction[b,c,d] (µg/kg)					k
			Wet-Mass Basis		Dry-Mass Basis			
PCB	8	(2,4'-Dichlorobiphenyl)	0.191 ±	0.003	1.86	±	0.03	1.97
PCB	18	(2,2',5-Trichlorobiphenyl)	0.589 ±	0.007	5.75	±	0.08	1.97
PCB	28	(2,4,4'-Trichlorobiphenyl)	1.47 ±	0.02	14.4	±	0.2	1.97
PCB	31	(2,4',5-Trichlorobiphenyl)	1.16 ±	0.06	11.3	±	0.6	1.97
PCB	44	(2,2'3,5'-Tetrachlorobiphenyl)	1.54 ±	0.08	15.1	±	0.8	1.97
PCB	45	(2,2',3,6-Tetrachlorobiphenyl)	0.214 ±	0.019	2.09	±	0.18	1.96
PCB	49	(2,2',4,5'-Tetrachlorobiphenyl)	1.76 ±	0.02	17.1	±	0.2	1.97
PCB	52	(2,2',5,5'-Tetrachlorobiphenyl)	2.49 ±	0.06	24.3	±	0.6	1.97
PCB	56	(2,3,3',4-Tetrachlorobiphenyl)	0.663 ±	0.008	6.46	±	0.09	1.98
PCB	63	(2,3,4',5-Tetrachlorobiphenyl)	0.137 ±	0.013	1.34	±	0.13	1.97
PCB	66	(2,3',4,4'-Tetrachlorobiphenyl)	1.65 ±	0.02	16.1	±	0.2	2.05
PCB	70	(2,3',4',5-Tetrachlorobiphenyl)	1.57 ±	0.05	15.3	±	0.5	1.97
PCB	74	(2,4,4',5-Tetrachlorobiphenyl)	0.850 ±	0.011	8.29	±	0.12	1.96
PCB	82	(2,2',3,3',4-Pentachlorobiphenyl)	0.507 ±	0.008	4.95	±	0.09	1.97
PCB	87	(2,2',3,4,5'-Pentachlorobiphenyl)	2.08 ±	0.02	20.3	±	0.2	2.01
PCB	92	(2,2',3,5,5'-Pentachlorobiphenyl)	1.06 ±	0.02	10.4	±	0.2	1.97
PCB	95	(2,2',3,5',6-Pentachlorobiphenyl)	1.82 ±	0.02	17.8	±	0.2	2.15
PCB	99	(2,2',4,4'5-Pentachlorobiphenyl)	3.55 ±	0.05	34.7	±	0.6	1.97
PCB	101	(2,2',4,5,5'-Pentachlorobiphenyl)	6.67 ±	0.05	65.1	±	0.7	1.97
PCB	105	(2,3,3',4,4'-Pentachlorobiphenyl)	1.57 ±	0.03	15.3	±	0.3	1.97
PCB	110	(2,3,3',4',6-Pentachlorobiphenyl)	5.47 ±	0.06	53.4	±	0.7	1.97
PCB	118	(2,3',4,4',5-Pentachlorobiphenyl)	4.08 ±	0.09	39.8	±	0.9	1.97
PCB	128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	0.801 ±	0.011	7.81	±	0.11	1.97
PCB	138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	4.39 ±	0.04	42.9	±	0.5	1.97
PCB	146	(2,2',3,4',5,5'-Hexachlorobiphenyl)	0.904 ±	0.005	8.82	±	0.09	1.97
PCB	149	(2,2',3,4',5',6-Hexachlorobiphenyl)	3.97 ±	0.04	38.8	±	0.5	1.97
PCB	151	(2,2',3,5,5',6-Hexachlorobiphenyl)	1.13 ±	0.03	11.0	±	0.3	1.96
PCB	153	(2,2',4,4',5,5'-Hexachlorobiphenyl)[a]	6.76 ±	0.12	66.0	±	1.3	1.97
	132	(2,2',3,3',4,6-Hexachlorobiphenyl)						
PCB	156	(2,3,3',4,4',5-Hexachlorobiphenyl)	0.253 ±	0.005	2.47	±	0.05	1.96
PCB	158	(2,3,3',4,4',6-Hexachlorobiphenyl)	0.443 ±	0.003	4.33	±	0.04	1.98
PCB	163	(2,3,3',4',5,6-Hexachlorobiphenyl)	1.10 ±	0.09	10.8	±	0.9	1.96
PCB	170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	0.105 ±	0.009	1.03	±	0.09	1.97
PCB	177	(2,2'3,3',4',5,6-Heptachlorobiphenyl)	0.696 ±	0.011	6.79	±	0.12	1.97
PCB	178	(2,2',3,3',5,5',6-Heptachlorobiphenyl)	0.350 ±	0.011	3.42	±	0.11	1.97
PCB	180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	0.594 ±	0.008	5.79	±	0.09	1.97
PCB	183	(2,2',3,4,4'5',6-Heptachlorobiphenyl)	0.848 ±	0.006	8.27	±	0.09	1.98
PCB	187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)	2.09 ±	0.05	20.4	±	0.5	1.97

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [8] and later revised by Schulte and Malisch [9] to conform with IUPAC rules; for the specific congeners mentioned in this table, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute under the GC analysis conditions used, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

[b] Mass fractions are reported on both wet- and dry-mass basis; material as received contains 89.75 % ± 0.08 % (95 % confidence level) water.

[c] The certified value reported on a wet-mass basis is a weighted mean of average mass fractions, with one average each from two analytical methods [3,4]. The expanded uncertainty is the half width of a symmetric 95 % parametric bootstrap confidence interval [5], which is consistent with the ISO Guide [6,7]. The effective coverage factor k is included in the table for each PCB congener.

[d] GC/MS (Ia) using SPE clean-up followed by analysis on a DB-17MS column using EI-MS and GC/MS (IIa) using SPE and SEC clean-up followed by analysis on a DB-XLB column using EI-MS.

Table 3. Certified Mass Fraction Values for Selected Chlorinated Pesticides in SRM 1974c

	Mass Fraction[a,b] (µg/kg)					k
	Wet-Mass Basis			Dry-Mass Basis		
Heptachlor[c]	0.132	± 0.006		1.29	± 0.06	1.97
cis-Chlordane[d]	1.20	± 0.05		11.7	± 0.5	1.97
trans-Chlordane[d]	0.741	± 0.013		7.23	± 0.14	1.97
cis-Nonachlor[d]	0.286	± 0.006		2.79	± 0.06	1.98
trans-Nonachlor[d]	0.742	± 0.005		7.24	± 0.07	1.97
Dieldrin[d]	0.285	± 0.021		2.78	± 0.20	1.97
2,4'-DDE[c]	0.346	± 0.002		3.38	± 0.04	1.98
4,4'-DDE[c]	1.85	± 0.02		18.1	± 0.2	1.99
2,4'-DDD[c]	0.398	± 0.004		3.88	± 0.05	1.96
4,4'-DDD[c]	1.30	± 0.09		12.7	± 0.8	1.97
2,4'-DDT[c]	0.942	± 0.027		9.19	± 0.27	1.97

[a] Mass fractions are reported on both wet- and dry-mass basis; material as received contains 89.75 % ± 0.08 % (95 % confidence level) water.

[b] The certified value reported on a wet-mass basis is a weighted mean of average mass fractions, with one average each from two analytical methods [3,4]. The expanded uncertainty is the half width of a symmetric 95 % parametric bootstrap confidence interval [5], which is consistent with the ISO Guide [6,7]. The effective coverage factor k is included in the table for each chlorinated pesticide.

[c] GC/MS (Ia) using SPE clean-up followed by analysis on a DB-17MS column using EI-MS and GC/MS (IIa) using SPE and SEC clean-up followed by analysis on a DB-XLB column using EI-MS.

[d] GC/MS (Ia) using SPE clean-up followed by analysis on a DB-17MS column using EI-MS and GC/MS (IIb) using SPE and SEC clean-up followed by analysis on a DB-XLB column using NCI-MS.

Table 4. Certified Mass Fraction Values for Selected PBDE Congeners[a] in SRM 1974c

			Mass Fraction[b,c,d] (µg/kg)					k
			Wet-Mass Basis			Dry-Mass Basis		
PBDE	17	(2,2',4-Tribromodiphenyl ether)	0.078	± 0.003		0.761	± 0.032	1.97
PBDE	25	(2,3',4-Tribromodiphenyl ether)	0.103	± 0.005		1.00	± 0.05	1.97
PBDE	47	(2,2',4,4'-Tetrabromodiphenyl ether)	0.939	± 0.017		9.16	± 0.18	1.96
PBDE	49	(2,2',4,5'-Tetrabromodiphenyl ether)	0.140	± 0.005		1.37	± 0.05	1.97
PBDE	99	(2,2',4,4',5-Pentabromodiphenyl ether)	0.375	± 0.004		3.66	± 0.05	1.97

[a] PBDE congeners are numbered according to IUPAC rules.

[b] Mass fractions are reported on both wet- and dry-mass basis; material as received contains 89.75 % ± 0.08 % (95 % confidence level) water.

[c] The certified value reported on a wet-mass basis is a weighted mean of average mass fractions, with one average each from two analytical methods [3,4]. The expanded uncertainty is the half width of a symmetric 95 % parametric bootstrap confidence interval [5], which is consistent with the ISO Guide [6,7]. The effective coverage factor k is included in the table for each PBDE congener.

[d] GC/MS (Ib) using SPE clean-up followed by analysis on a DB-17MS column using NCI-MS and GC/MS (IIa) using SPE and SEC clean-up followed by analysis on a DB-XLB column using EI-MS.

Table 5. Reference Mass Fraction Values for Selected PAHs in SRM 1974c

	Mass Fraction[a] (µg/kg)						k
	Wet-Mass Basis			Dry-Mass Basis			
Naphthalene[b,c]	0.990	±	0.039	9.66	±	0.39	1.96
1-Methylnaphthalene[b,c]	1.41	±	0.03	13.7	±	0.3	1.97
2-Methylnaphthalene[b,c]	1.50	±	0.06	14.6	±	0.6	1.97
1,2-Dimethylnaphthalene[b,c]	0.913	±	0.007	8.91	±	0.10	2.09
1,6-Dimethylnaphthalene[b,c]	1.19	±	0.02	11.6	±	0.2	1.97
2,6-Dimethylnaphthalene[b,c]	0.206	±	0.006	2.01	±	0.06	1.97
Biphenyl[b,c]	0.860	±	0.008	8.39	±	0.10	1.97
Acenaphthylene[b,c]	0.523	±	0.007	5.11	±	0.08	1.96
Acenaphthene[b,c]	0.343	±	0.019	3.35	±	0.18	1.97
1-Methylfluoranthene[d,e]	0.451	±	0.014	4.40	±	0.14	2.57
3-Methylfluoranthene[d,e]	1.32	±	0.02	12.9	±	0.2	2.57
Chrysene[d,e]	19.2	±	0.5	187	±	5	2.09
Triphenylene[d,e]	10.1	±	0.1	98.5	±	1.6	2.09
4-H-Cyclopenta[def]phenanthrene[d,e]	2.02	±	0.04	19.7	±	0.4	2.57
Benzo[b]fluoranthene[d,e]	5.95	±	0.05	58.0	±	0.7	2.09
Benzo[j]fluoranthene[d,e]	2.07	±	0.01	20.2	±	0.2	2.09
Dibenz[a,c+a,h]anthracene[d,e]	0.100	±	0.001	0.976	±	0.016	2.57
Dibenzo[b,k]fluoranthene[d,e]	0.490	±	0.010	4.78	±	0.11	2.57

[a] Mass fractions are reported on both wet- and dry-mass basis; material as received contains 89.75 % ± 0.08 % (95 % confidence level) water.

[b] GC/MS (Ia) using SPE clean-up followed by analysis on a DB-17MS column using EI-MS and GC/MS (IIa) using SPE and SEC clean-up followed by analysis on a DB-XLB column using EI-MS.

[c] The reference value reported on a wet-mass basis is a weighted mean of average mass fractions, with one average each from two analytical methods [3,4]. The expanded uncertainty is the half width of a symmetric 95 % parametric bootstrap confidence interval [5], which is consistent with the ISO Guide [6,7]. The effective coverage factor k is included in the table for each PAH.

[d] GC/MS (IIa) using SPE and SEC clean-up followed by analysis on a DB-XLB column using EI-MS.

[e] The reference value reported on a wet-mass basis is the mean of results obtained using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's t-distribution corresponding to the associated degrees of freedom and a 95 % confidence level for each analyte.

Table 6. Reference Mass Fraction Values for Selected PCB Congeners[a] in SRM 1974c

			Mass Fraction[b,c,d] (µg/kg)						k
			Wet-Mass Basis			Dry-Mass Basis			
PCB	29	(2,4,5-Trichlorobiphenyl)	0.131	±	0.003	1.28	±	0.03	2.57
PCB	109	(2,3,3',4,6-Pentachlorobiphenyl)	0.451	±	0.004	4.40	±	0.06	2.57
PCB	114	(2,3,4,4',5-Pentachlorobiphenyl)	0.155	±	0.005	1.51	±	0.05	2.57
PCB	119	(2,3',4,4',6-Pentachlorobiphenyl)	0.341	±	0.004	3.33	±	0.05	2.57
PCB	130	(2,2',3,3',4,5'-Hexachlorobiphenyl)	0.356	±	0.008	3.47	±	0.08	2.57
PCB	137	(2,2',3,4,4',5-Hexachlorobiphenyl)	0.095	±	0.001	0.924	±	0.014	2.57
PCB	154	(2,2',4,4',5,6'-Hexachlorobiphenyl)	0.990	±	0.020	9.66	±	0.22	2.57
PCB	157	(2,3,3',4,4',5'-Hexachlorobiphenyl)	0.086	±	0.003	0.840	±	0.026	2.57
PCB	165	(2,3,3',5,5',6-Hexachlorobiphenyl)	1.56	±	0.02	15.2	±	0.3	2.57
PCB	166	(2,3,4,4',5,6-Hexachlorobiphenyl)	0.020	±	0.001	0.192	±	0.010	2.57
PCB	167	(2,3',4,4',5,5'-Hexachlorobiphenyl)	0.305	±	0.004	2.98	±	0.05	2.57
PCB	175	(2,2',3,3',4,5',6-Heptachlorobiphenyl)	0.139	±	0.002	1.36	±	0.03	2.57
PCB	176	(2,2',3,3',4,6,6'-Heptachlorobiphenyl)	0.165	±	0.004	1.61	±	0.04	2.57
PCB	202	(2,2',3,3',5,5',6,6'-Octachlorobiphenyl)	0.214	±	0.003	2.09	±	0.04	2.57

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [8] and later revised by Schulte and Malisch [9] to conform with IUPAC rules; IUPAC PCB 109 is BZ#107.

[b] Mass fractions are reported on both wet- and dry-mass basis; material as received contains 89.75 % ± 0.08 % (95 % confidence level) water.

[c] The reference value reported on a wet-mass basis is the mean of results obtained using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's t-distribution corresponding to the associated degrees of freedom and a 95 % confidence level for each analyte.

[d] GC/MS (IIa) using SPE and SEC clean-up followed by analysis on a DB-XLB column using EI-MS.

Table 7. Reference Mass Fraction Values for Selected Chlorinated Pesticides in SRM 1974c

	Mass Fraction[a,b,c] (µg/kg)						k
	Wet-Mass Basis			Dry-Mass Basis			
Hexachlorobenzene	0.021	±	0.001	0.205	±	0.014	2.57
Mirex	0.164	±	0.005	1.60	±	0.05	2.57

[a] Mass fractions are reported on both wet- and dry-mass basis; material as received contains 89.75 % ± 0.08 % (95 % confidence level) water.

[b] The reference value reported on a wet-mass basis is the mean of results obtained using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's t-distribution corresponding to the associated degrees of freedom and a 95 % confidence level for each analyte.

[c] GC/MS (IIb) using SPE and SEC clean-up followed by analysis on a DB-XLB column using NCI-MS.

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